Available online at www.sciencedirect.com

ScienceDirect

Physics Procedia 78 (2015) 305 – 311

Physics Procedia

15th Nordic Laser Materials Processing Conference, Nolamp 15, 25-27 August 2015, Lappeenranta, Finland

# Local Reversion of Cold Formed AISI 301LN

A. Järvenpää[a]*, M. Jaskari[a], M. Hietala[a], K. Mäntyjärvi[a]

[a]*University of Oulu, Oulu Southern Institute, Pajatie 5, FIN-85500 Nivala, Finland*

**Abstract**

This study demonstrates applying laser heat treatment for reversion treatments of cold-formed AISI 301LN. Sheets were cold-rolled to final thicknesses of 1.5 and 3 mm (65pct reduction), having martensite fraction of 70-95%. Sheets were heated locally by a laser beam to various peak temperatures to obtain different degrees of martensite reversion to austenite. Mechanical properties and formability of grain-refined and coarse-grained structures were measured by tensile, bending and Erichsen cup tests. In addition to standard Erichsen cup test, additional interrupted tests were carried out, where cups were first stretched close to the critical strain. Drawn cups were then heated locally by a laser beam to revitalize the structure and thereby enhance the formability in the following cupping test until failure.

Various structures were produced: completely reverted microstructures (T > 700 °C) with grain sizes 0.9 - 2 μm in addition to partially reverted structure (T < 700 °C) containing nano- and ultrafine-grained austenite (0.6 μm) with some martensite. Results showed that local laser heat treatment is suitable for the reversion treatment to refine the austenite grain size. Refinement of the austenitic structures increased strength properties and the formability was better than with coarse grained structures having the same strength. Especially the yield strength was significantly enhanced, being around 900 MPa in the strongest reverted structure compared to the 300-400 MPa of the coarse grained austenitic structure. It was demonstrated that the local laser treatment restored formability of the drawn cups, allowing stretching to be continued.


Peer-review under responsibility of the Lappeenranta University of Technology (LUT)

*Keywords:* Laser; Local heat treatment; Reversion; Strain hardening

* Corresponding author. Tel.: +358405551633
*E-mail address:* antti.jarvenpaa@oulu.fi


Peer-review under responsibility of the Lappeenranta University of Technology (LUT)
doi:10.1016/j.phpro.2015.11.041

## 1. Introduction

Austenitic stainless steels are widely used in industrial and domestic applications, due to its ductility and corrosion resistance. Tensile strength, however, tends to be quite low in annealed structures, about 300 MPa. Cold forming increases the yield and tensile strengths, but also diminishes the ductility properties. An effective method for increasing the mechanical properties of austenitic stainless steels is to refine the austenite grain size (GS) (Hall-Petch relation) as shown in previous studies e.g. by Schino (2002) and Somani (2007), that refining the grain size to nano- or submicron size enhances the tensile properties without impairing the ductile properties. Obtained yield strengths have almost been doubled, when compared to commercial SS grades in annealed conditions. An effective method to obtain the ultra-fine grained austenite (UFGA) structure is so called reversion treatment, where a heavily cold formed metastable stainless steel containing large amounts of martensite is heat treated under the austenization temperature (650–950 °C). In 301LN, for example, the heat treatment and high heating rate enables the strain-induced martensite to revert back to austenite through diffusion mechanisms as shown e.g. by Somani (2009) and Rajasekhara (2010). Recent studies by Järvenpää et al (2014) have been focusing on to produce so called partially reverted structures where deformed austenite (from cold-rolling) and tempered martensite are also present in the UFGA structure. These partially reverted structures have shown a great potential, especially due to very high yield strength and good formability.

In this study, the aim was to implement a local laser heat treatment (LLHT) to revert the cold-rolled structure of AISI 301LN back to ultra-fine grained austenite, and further to enhance the formability. Järvenpää et al studied the suitability of a LLHT for tailoring mechanical properties of different steel grades. In 2009 Järvenpää et al studied the effect of sheet thickness on laser hardening. During these experiments, 1.8 mm thick boron steel was successfully hardened through the cross-section. In 2012, the LLHT method was successfully adapted for softening hard steel grades up to thickness of 10 mm by Järvenpää et al. First trials on local reversion were carried out in 2012-13 using the same experimental methods. In addition to the main objective of the study (suitability of LLHT for different stages of reversion), it is interesting to speculate the possibilities for a two steps forming operations were reversion is utilized after the first forming step to revitalize the microstructure (expanded forming limit) and to produce excellent static and dynamic strength properties for the final product. This study presents the results from LLHT reversion studies. Cold-forming for the LLH-tests were made using two different cold-rolling parameters and Erichsen drawing tests to produce strain induced martensite.

## 2. Experimental Methods

### *2.1. Test Material*

Table 1. Nominal chemical composition in wt%.

| Material | C | Mn | Si | Cr | Ni | Mo | N | Fe |
|---|---|---|---|---|---|---|---|---|
| AISI 301LN | 0.017 | 1.29 | 0.52 | 17.3 | 6.5 | 0.15 | 0.15 | bal. |

The test material used was an austenitic, commercial Cr-Ni stainless steel AISI 301LN, which chemical composition determined by a glow discharge optical emission spectrometer (GDA 750) was as follows (in wt%): 0.018C, 1.12Mn, 0.48Si, 17.9Cr, 6.3Ni, 0.08Mo and 0.12N. Three versions of the material were used in this study: 1) as-delivered (t = 3 mm) 10% cold-rolled (temper rolled) corresponding to the C700 grade, 2) a 65% cold-rolled together with liquid nitrogen cooling (t = 3 mm) with the martensite fraction of over 90% and 3) a thinner (t = 1.5 mm) version of the cold rolled steel was used in bending tests, martensite fraction being lower, about 70% (rolling without additional cooling).

Table 2. Test material properties.

| Material | Reduction [%] | Thickness [mm] | Martensite fraction [%] | Hardness [HV1] |
|---|---|---|---|---|
| AISI 301LN CR1 | 14+50 | 1.5 | 70 | 560 |
| AISI 301LN CR2 | 6+50 | 3.0 | 95 | 585 |
| AISI 301LN C700 | 6 | 3.0 | 1-2 | 250 |

*2.2. Local laser Heat Treatments (LLHT)*

Pieces of the cold-rolled sheets were locally heat treated by a laser beam using various heating parameters to demonstrate the effect of new reverted grain size on mechanical properties. In this study, the laser employed was a diode pumped 4 kW Yb:YAG (Trumpf HDL 4002) with a Precitec YW50 welding head. Movement of the laser beam was controlled by a Motoman UP50N robot. Scanning of the beam was done to specimen top surface with constant amplitude of 16 mm in 3 mm sheets, and 8 mm in 1.5 mm sheet with linear movement, as shown in Fig. 1. The maximum laser power of 4 kW and the distance from the focal point to the surface of the workpiece (focus distance) of +185.5 mm (spot size of 19.78 mm) were held in each experiment, and maximum temperature was varied by changing the linear speed. Temperature was measured using K-type thermocouples. Thermocouples were fixed on the middle of the laser-treatment scan (bottom surface), as shown in Fig. 1. The aim of the laser-treatments was to produce the completely reverted (Rev) microstructures (T > 700 °C) with various grain sizes, as well as to obtain a partially reverted structure (T < 700 °C) containing nano- and ultrafine-grained austenite with some martensite.

Table 3. LLHT processing parameters.

| Material | Scanning Amplitude [mm] | Linear Speed [mm/s] |
|---|---|---|
| CR1 | 8 | 14 |
| | | 15 |
| | | 16 |
| | | 16.5 |
| | | 18.5 |
| | | 20 |
| CR2 | 16 | 4.8 |
| | | 5.5 |
| | | 5.8 |

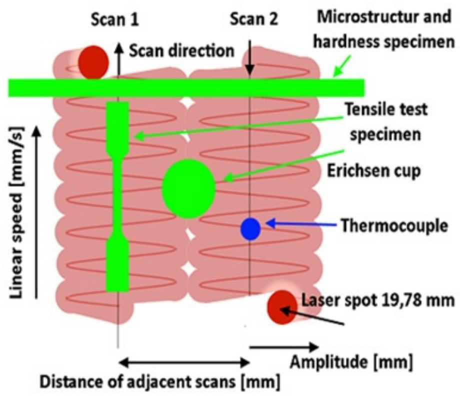

Fig 1. Laser processing principle.

*2.3. Erichsen cup tests.*

Pieces of 90 x 500 mm were cut from the sheets. Erichsen cups were stretched (at Kemi-Tornio University of Applied Sciences) from the as-delivered C700 sheet pieces as well as from laser-reverted CR2 sheet pieces with various reverted structures by using an Erichsen universal sheet metal testing machine, Model 145-60 (the maximum force 400 kN, retaining force 10 kN, the punching speed 10 mm/min, graphite grease lubricant; the indenter diameter 20 mm and Erichsen test nr. 40). Cup test were done in two ways: the maximum cup depth for the conventional coarse grained austenitic (CGA) and the ultra-fine grained austenitic (UFGA) structures were first determined and then an interrupted punch test, where the punch was stopped before the breaking of the sheet. Cups were then revitalized using local laser heat treatment, and a second punch was applied until specimen fracture.

*2.4. Bending tests*

Bending tests were done at University of Oulu, and Ursviken Optima M-4721 bending machine was used to bend the samples to 90 degrees. Used upper punch tool had radius of 1 mm, being the smallest obtainable, and used lower die was W45. Tests were done for every heat treatment parameter until a limit value was found, and from each test bending forces were measured. Tests were recorded using HD video camera for springback calculations.

### *2.5. Mechanical Properties*

Tensile tests were performed using a Zwick100 tensile test machine equipped with an extensometer. The tensile test specimen dimensions were according to the standard EN 10002-1. Hardness of the various structures was measured using a Struers Duramin A300 hardness tester with the Vickers indenter (HV1). The hardness of both the upper and bottom surfaces of the cup specimens was measured to record the difference between the surfaces. Hardness profiles were also determined across the laser-heat treated sheets.

### *2.6. Metallographic studies*

Optical microstructures were examined with a Keyence VK-X200 series laser microscope to achieve high-resolution scans across the specimen, using an UV-laser. A Ferritescope (Fischer MP30) instrument was used to measure the martensite fraction in the cup test specimens, ten times for each test location to minimize the statistical error. The obtained readings were multiplied by 1.7 to obtain martensite fractions, as suggested by Talonen et al. (2005).

## 3. Results

### *3.1. Laser Heat Treatments*

Laser heat treatments were conducted in three parts, where first one was done to material CR1. Main goal was to find desired heating parameters for complete martensite reversion to UFGA. The desired temperature range between 700 °C and 900 °C was reached with approximately 10 seconds in the austenite region ("holding time"), and as can be seen form Tables 4 and 5, the hardness decreased as the peak temperature increased. This can be explained by reversion driven by diffusion, where formed UFGA grew due the greater heat input, which can also be seen from Fig. 2. Linear speeds varied from 14 mm/s to 20 mm/s in CR1, and from 4.8 to 5.8 mm/s when heat treating the material CR2.

Overall, the microstructure was austenitic, with some retained austenite (RA), which had persisted from cold rolling. In some large cold-rolled austenite grains, partially recrystallized shear bands were frequently present. Also some areas contained some amount of martensite due partial reversion. Structures were similar than observed in earlier studies, although the single laser beam caused some deviation between the surfaces.

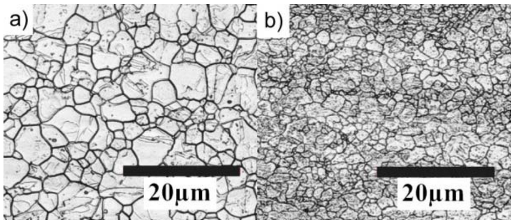

Fig. 2. Reverted coarse grained austenite, T = 850 °C (a), and ultrafine-grained austenite T = 750 °C.

Table 4. Hardness and mechanical properties of CR1 with different heating temperatures.

| Temperature [°C] | Linear Speed [mm/s] | Hardness [HV1] | Yield Strength [MPa] | Tensile Strength [MPa] | UE [%] | TE [%] |
|---|---|---|---|---|---|---|
| RT | - | 560 | 1440 | 1600 | 0.6 | 1.6 |
| 900 | 14 | 230 | 450 | 800 | 44 | 48.5 |
| 850 | 15 | 280 | 565 | 840 | 37.3 | 41.9 |
| 800 | 16 | 290 | 560 | 840 | 38.2 | 43.1 |
| 750 | 16.5 | 315 | 798 | 935 | 24.9 | 29.5 |
| 700 | 18.5 | 383 | 920 | 1015 | 12 | 18.2 |
| 650 | 20 | 475 | 972 | 1055 | 8.9 | 12.9 |

Austenite grains sizes depend on the peak temperatures. The most coarse GS was found from highest heating temperatures, being around 10 µm, and the finest, from 1 to 2 µm, from the lowest heating temperatures, respectively. Grain size distribution also varied between the surfaces, due to austenite low thermal conductivity and the fact, that laser treatment was applied only to top surface. This emphasized most profoundly in temperatures fewer than 700 °C, when the bottom surface was left partly martensitic. The smallest austenite grains were under 200 nm (Fig. 3.), found as islands between martensite matrix.

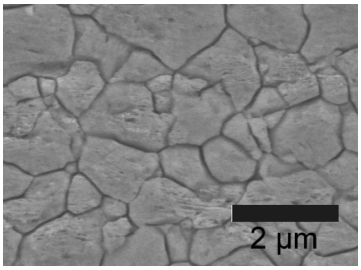

Fig 3. Reverted austenite structure, SEM-image, 15 000x zoom. T = 720 °C

Table 5. Hardness and mechanical properties of AISI 301LN C700 and laser heat treated AISI 301LN CR2.

| Temperature [°C] | Linear Speed [mm/s] | Hardness [HV1] | Yield Strength [MPa] | Tensile Strength [MPa] | UE [%] | TE [%] |
|---|---|---|---|---|---|---|
| AISI 301LN C700 | - | 250 | 538 | 861 | 34.5 | 39.5 |
| T = 800 | 4.8 | 296 | 605 | 860 | 35.1 | 41.7 |
| T = 700 | 5.5 | 370 | 778 | 963 | 27.9 | 33.2 |
| T = 650 | 5.8 | 416 | 858 | 994 | 11.7 | 24.1 |

### *3.2. Bending*

Bending tests were done for the CR1 in University of Oulu, with Ursviken Optima M-4721 bending machine. Acquired bending forces and springbacks were measured, and as can be seen in Table 6. When temperature was over 700 °C, bending was successful with the smallest upper tool, radius being 1mm. This was due to completed reversion treatment, as austenite has very good ductility properties. Bending forces increased, when the treatment temperatures decreased due to the austenite grain size refinement, smallest GS being below 2 µm. When heat treatment temperature was 700 °C and under, reversion didn't complete throughout the full thickness of the sheet, leaving the bottom surface partly martensitic. This caused impaired ductility, and some of the samples fractured before desired bending angle. The limit value in bending was found at temperature area of 750 °C, when at least one of the samples fractured. Bending process is shown in Fig. 4. Bending forces and springbacks increased, as the material strength increased, from 146 kN/m with LLHT treated in 900 °C to 169 kN/m in LLHT treated in 650 °C. The acquired bending ratio R/t was 0.67, which was considered as a good indicator for excellent bendability.

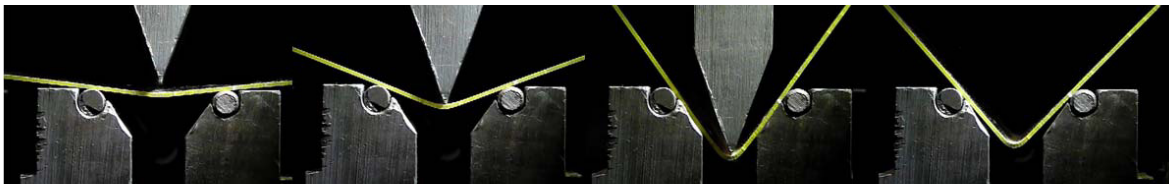
Fig. 4. Schematic picture of the bending process.

Table 6. Bending test results.

| Temperature [°C] | Measured Force [kN/m] | Bending Angle | Backspring |
|---|---|---|---|
| 900 | 146 | 88.8 | 16.4 |
| 850 | 151 | 89.6 | 17.5 |
| 800 | 154 | 89.6 | 17.4 |
| 650 | 169 | 88.2 | 19.0 |

### 3.3. Erichsen cup tests.

Cupping tests were done to measure the deep drawing properties of the reverted structures. Used test material was AISI 301LN CR2, laser heat treated with parameters explained earlier in section 3.1. Thicker sheet was considered necessary, as more plastic deformation could be applied. Three heat treatment temperatures were selected for the formability study: 800 °C for UFGA structure, 700 °C for a very fine austenitic structure and 670 °C for partially reverted structure. Holding times were almost equal, being around 10 – 15 s for each parameter. 3 mm thick commercial AISI 301LN C700 was used as reference

First punches were done to measure the formability of heat treated samples. Forming forces and maximum cup depths can be seen from Table 7, it can be concluded that although maximum punch forces were almost identical, the evolution of the force was different when compared to commercial reference specimen. This was partly due to austenite grain refinement in LLHT specimen treated in 800 and 900 °C. The martensite formation paths and kinetics are different when grain size (GS) is very small. Below 2 μm GS, martensite transformation takes place at grain boundaries making the phenomena quicker than in coarse grained structures where martensite transformation takes place via slip band formation as observed in earlier studies. The difference in strain hardening rate can be seen in Fig. 5 where reverted structures are on left side of the reference structure. Further, in partly reverted structure the presence of martensite increased the needed punch forces, but also impaired the ductility, decreasing the cup depth to 12.4 mm. Cup depths of AISI 301LN C700, LLHT specimen treated at 800 C and 700 C were 16.2, 16.4 and 16.0 mm, respectively.

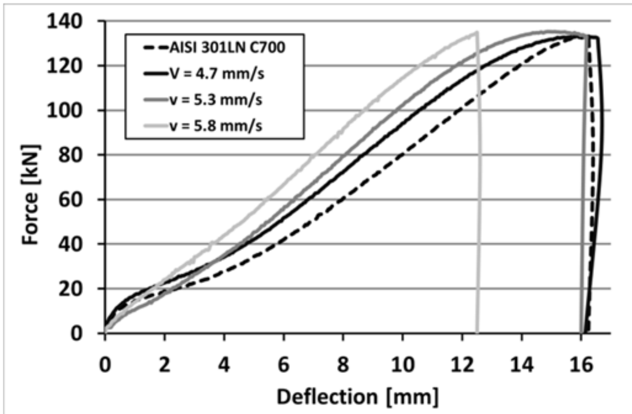


Fig. 5. Maximum cup depths and forces

Second punch was applied to revitalized cups, which were interrupted at punch depth of 12 mm. Only the reference material and LLHT specimen treated in 800 °C were used, because the purpose of the test was to demonstrate the idea of repeating the LLHT to formed areas. Although the idea was to use reversion treatment, difficulties in parameter adjusting caused the temperatures rise above the austenization temperature, and so the treatment could be considered as recrystallization treatment. Acquired grain sizes were coarse, but the final punch depth could be still enhanced, up to 19%, as can be seen from Table 8. Final punch depths before fracture were 19.4 mm for AISI 301LN C700 and 18.7 mm for specimen treated at 800 °C, respectively. Due to the improper heat distribution, the final martensite fractions were decreased.

Table 7. Erichsen test results.

| Description | Maximum Punch Depth [mm] | Maximum Punch Force [kN] | Max. Martensite Fraction [%] | Max. Hardness [HV] |
|---|---|---|---|---|
| AISI 301LN C700 | 16.2 | 134 | 83 | 527 |
| T = 800 °C | 16.4 | 133 | 93 | 552 |
| T = 700 °C | 16.0 | 135 | 93 | 563 |
| T = 670 °C | 12.4 | 134 | 100 | 546 |

Table 8. Test results of the second punch.

| Description | Punch 1 [mm] | Punch 2 [mm] | Original Max. Punch Depth [mm] | Enhancement [%] | Max. Punch Force [kN] | Max. Martensite Fraction [%] | Max. Hardness [HV] |
|---|---|---|---|---|---|---|---|
| 301LN C700 | 12 | 19.4 | 16.3 | 19 | 99 | 63 | 511 |
| T=800 °C | 12 | 18.7 | 16.4 | 14 | 112 | 79 | 535 |

## 4. Conclusions

The present study showed that the local laser heat treatment (LLHT) was successfully adapted to revert cold-rolled AISI 301LN stainless steel. Study was made in two phases using two different cold-rolled 301LN batch having different martensite fractions (70% and 95%). In both cases, excellent mechanical properties were achieved supporting earlier observations e.g. by Somani (2009). Study was a straight continuum for author's earlier work with ferritic steel grades where excellent properties and stable processing was observed. The recent study supports the fact that laser processing is actually very repeatable and also suitable for heat treating relatively thick steel sheets.

Local reversion can be used e.g. to soften cold-formed metastable austenitic steel grades e.g. as a pretreatment before forming. Method can also be applied to expand the forming limits. Forming operation can be interrupted before crack initiation and then be reverted locally using laser or induction heating to revitalize the microstructure. Forming can then be begun again from annealed structure having excellent strength and forming properties. Empirical tests on LLHT of a formed Erichsen cup were not completely successful due to lack of material that narrowed the parameter tuning. Due to the thinning of the cup wall and the height of the cup, too high peak temperatures were produced and the transformation mechanism was actually recrystallization instead of reversion. Bending tests did not reveal any significant differences in bendability, because of a great formability of austenitic steel grades. Results indicate that the UFGA structure has similar formability than CGA structure even though the yield strength in UFGA is significantly higher.

## Acknowledgements

The financial support from Tekes for the "ULLA" project and from the Council of Oulu Region as well as the European Union (European Regional Development Fund) for the "RPST" project is acknowledged. The authors would especially like to thank companies Miilux Oy and SSAB for the test materials and their continuous support during the research.

## References

Di Schino, A., Barteri, M., J.M. Kenny, J.M., 2002, "Development of ultra-fine grain structure by martensitic reversion in stainless steels", Journal of Materials Science Letters 21, 2002, p.751-753

Järvenpää, A., Karjalainen, L.P., Mantyjarvi, K., 2012, "Passive laser assisted bending of ultra-high strength steels", Adv. Mater. Res. 418-420 (2012) 1542-1547

Järvenpää, A., Karjalainen, L.P., Jaskari, M., 2014, "Effect of Grain Size on Fatigue Behaviour of Type 301ln Stainless Steel" International Journal of Fatigue Vol 65 (2014), ISSN 0142-1123

Järvenpää, A., Jaskari, M., Karjalainen, L.P., Hietala, M., 2013, "Enhancing mechanical properties and formability of AISI 301LN stainless steel sheet by local laser heat treatment", Key Engineering Materials, The Current State-of-the-Art on Material Forming (2013) ISBN-13: 978-3-03785-719-9

Mäntyjärvi, K., Karjalainen, J.A., Järvenpää, A., Ojala, J., Keskitalo, M., Mäkikangas, J., 2009, "Water Cooling in Sheet Metal Laser Hardening", Proceedings of the 12th NOLAMP Conference in Laser Proc-essing of Materials, 24th-26th of August 2009, Copenhagen, Denmark (2009)

Rajasekhara, S., Karjalainen, L.P., Kyröläinen, A., Ferreira, P.J., 2010, "Microstructure evolution in nano/submicron grained AISI 301LN stainless steel". Mater Sci Eng A 2010;527:1986–96.

Somani, M.C., Karjalainen, L.P., Kyrolainen, A., Taulavuori, T., 2007, "Processing of submicron grained microstructures and enhanced mechanical properties by cold-rolling and reversion annealing of metastable austenitic stainless steels, Mater. Sci. Forum 539-543 (2007) 4875-4880

Somani, M.C., Juntunen, P., Karjalainen, L.P., Misra, R.D.K., Kyrolainen, A., 2009, "Enhanced mechanical properties through reversion in metastable austenitic stainless steels", Metall. Mater. Trans. A 40A (2009) 729-744

Talonen, J., Nenonen, P., Pape, G., Hanninen H., 2005, "Effect of strain rate on the strain-induced $\gamma \rightarrow \alpha'$ martensite transformation and mechanical properties of austenitic stainless steels". Metall. Trans. A 36 (2005) 421–32